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(54) Title: PROCESS FOR BIOTECHNICAL FINISHING OF WOOL

(57) Abstract: The present invention relates to a method for finishing woollen textiles. According to the method, the woollen textile in an aqueous solution is brought into contact with protease enzyme in a large amount of water so that the woollen textile is moved as little as possible or not at all, at a temperature of about 60°C or less for 10 to 90 min. The enzyme is inactivated and the woollen textile is taken to dry in mechanical drying at a temperature of about 60°C or less to reach residual moisture content of 10 to 45 %, and the final drying is carried out without mechanics.

## PROCESS FOR BIOTECHNICAL FINISHING OF WOOL

The present invention relates to the method according to the preamble of claims 1 and 2 to a method for finishing woollen textiles, and to a woollen textile according 5 to the preamble of claim 18.

The heat insulation and moisture absorption properties of knitted and woven woollen fabrics and any garments produced from them are considerably better than those of the products made of synthetic fibres or other natural fibres. However, one problem with these products is the scale structure of the wool fibre. When wool is 10 washed with water and subjected to mechanical action, a directional friction effect, which is connected with the structure of the scales incorporated into the wool fibre itself, causes felting and shortening of the wool fibre; as a consequence, the wool article shrinks and, in addition, the wool article feels rough because of the scales. Various treatments have been developed in trying to prevent the shrinkage of wool, 15 which processes are described, e.g., in the US patent No. 5,980,579: efforts have been made (1) to modify the scale structure of the wool fibre in such a way so as to reduce or eliminate the directional friction effect, (2) to increase the number of intermolecular cross-links so as to decrease the elasticity of the fibres, (3) to cover the fibres with a thin film which masks the surface, thereby removing the cause of the 20 directional friction effect; and (4) to cement the fibres with a polymer that stiffens the structure so that no dimensional changes are able to take place.

To prevent shrinkage in wool processing, chlorine based compounds have been used, including gaseous chlorine, sodium hypochlorite, dichloroisocyanuric acid or potassium permanganate in conjunction with hypochlorite at a high pH, or sodium 25 sulphate or permonosulphuric acid at a low pH. When using a polymer, hexamethylenediamine has been used in a first stage, and sebacyl chloride in a second stage. A processing method of wool most commonly used is the IWS/CSIRO Chlorine Hercosett method, wherein an acid chlorination is followed by a treatment with the polymer.

30 Other finishing methods suggest treating wool with enzymatic processes, e.g., protease (US 6,258,129) in conjunction with plasma treatment, with an oxidase or a peroxidase solution (WO 98/27264), or by using peroxidase, catalase or lipase (US 5,529,928) or a proteolytic enzyme in conjunction with transglutaminase (WO 99/60200) or the proteolytic enzyme and haloperoxidase in conjunction with a 35 source of hydrogen peroxide and a source of halide (WO 99/60199). Treating keratin-containing material with an alkali-containing alcohol solution and a protease-

containing aqueous solution has also been suggested (WO 00/50686). The published patent application WO 99/42649 treats wool in water with subtilisin protease in a large amount of water at various temperatures. Finally, the wool is treated by means of wet waxing.

5 However, in the enzymatic finishing methods presently used, about 50% of the strength of the wool fibre is lost. If the tips of the scales are filled chemically with various resins, for example, the effect will cease as soon as after the first water washing cycle, whereby the product turns rough and fuzzy. Furthermore, wool articles finished with the techniques presently used shrink about 10 – 15% when  
10 washed with water, whereas the goal is below 3%.

As woollen articles do not withstand washing with water, the manufacturers of the articles must recommend dry cleaning for the articles, which, in turn, renders the use of woollen articles more expensive and more difficult to the consumer. For all  
15 the above reasons, woollen articles are not widely used, although the good properties of woollen articles are commonly known.

The purpose of the present invention is to provide solutions, which can be used to improve the finish of woollen textiles so as to maintain the good properties of the same. The purpose, in particular, is to provide solutions, which can be used to make woollen textiles resistant to abrasion and water wash without shrinking, felting or  
20 pilling, and to maintain the good properties, such as warmth and excellent appearance, as well as the strength properties. A further purpose is to provide solutions, which can be used to make the woollen textiles feel softer.

In connection with the present invention, it was surprisingly observed that no single factor, such as the enzyme or the chemical used, had any effect on how to satisfactorily finish the woollen articles. Instead, the entire finishing process of wool must be set up so that the wool fibres are not subjected to excessively high temperatures or mechanical strain in an inappropriate stage. The wool fibre or the textiles containing wool fibres are particularly vulnerable during the wet process.

According to an advantageous embodiment of the invention, woollen textiles are  
30 treated using a method, wherein the woollen textile in an aqueous solution is brought into contact with protease in a large amount of water so that the woollen textile is moved as little as possible or not at all.

The temperature should be about 60°C or less. The treatment time can be about 10 to 90 min. After this, the enzyme is inactivated. After the protease treatment, the  
35 woollen textile is taken to dry in mechanical drying method so as to reach residual

moisture content of about 10 to 45%. The final drying of the woollen textile is preferably carried out without any mechanics, e.g., flat or in a hanging form.

More specifically, the method according to the invention is mainly characterized by what is stated in the characterizing part of claim 1. The woollen textile according to 5 the invention is mainly characterized by what is stated in the characterizing part of claim 18.

According to a preferred embodiment of the invention, the enzyme is inactivated by 10 using a method, wherein the temperature is not raised to above 60°C. Decreasing the pH sufficiently low or, for example, by adding copper ions may inactivate the enzyme. On the other hand, wool endures even higher temperatures, when the pH is made sufficiently low. Therefore, according to another preferred embodiment of the invention, the enzyme is inactivated by means of a method, wherein the pH is lowered to a range of about pH 4 to 5 or less.

Dyeing can also be combined with the treating method of woollen textiles. Dyeing 15 is preferably carried out in connection with the wet process. If dyeing is carried out after the enzyme treatment, the enzyme does not need a separate inactivation but the enzyme can be allowed to inactivate under the dyeing conditions.

More specifically, the method according to the invention, which dyeing is incorporated into, is characterized by what is stated in the characterizing part of claim 2.

20 When processing wool fibres according to the present invention, a substantial part of the edges of the tips of the wool fibre scales can be removed enzymatically. This causes some decrease in weight during treatment but, as a whole, the treatment causes no essential weakening of the strength properties of woollen textiles, which is the case in previously known enzymatic finishing methods, as in them, both desired and undesired parts come off the fibres uncontrollably because of the heavy 25 enzymatic and mechanical treatments. Also the mechanical drying phase, such as the tumble-drying stage, normally causes a decrease in strength, but in this method, the mechanical drying stage is kept so short that no essential decrease in strength takes place. Mechanical drying is continued to certain residual moisture content 30 only, after which the final drying is preferably carried out without mechanics, for example, flat or in a hanging form.

The purpose of the previously known methods was to prevent the shrinkage of wool by means of different chemical and enzymatic methods, but no previous method has 35 enabled the manufacture of woollen textiles that resist successive water wash without shrinking and pilling. Furthermore, the solutions according to the known tech-

nology have generally been implemented on a laboratory scale only, whereas the present invention is implemented on an industrial scale.

It has been observed that, in water wash, the woollen textiles, which were treated with the present invention, shrank 3% or less from the original. It was further observed that the textiles retained their properties for at least five or as many as 20 successive washings.

The finish according to the present invention can be carried out on dyed or undyed woollen textiles. According to a preferred embodiment of the invention, the woollen textile is first subjected to enzyme treatment and then to dyeing. It has been observed that extremely good dyeing results are obtained in this way. The colours have been found to be brighter and deeper, when dyeing is carried out after the enzyme treatment.

Woollen textiles are mainly made by using the worsted or the carded wool yarns methods. In the worsted method, long-stapled wool is used, from which lightweight or medium-heavy woven and knitted fabrics are made. Examples of worsted articles include suiting, trousering and light knitted fabrics. Woollen textiles can be dyed as tops, yarn, fabric or ready-made articles.

For the carded wool yarns, short-staple wool is used, from which heavy weight woven and knitted fabrics are made. Examples of products made by using the carded wool yarns include upholstery fabrics, thick knitted fabrics, felts, flannels and tweeds. These woollen textiles are dyed as fibre, yarn, fabric or finished articles.

“The finish of woollen textiles” refers to any procedure that can be used to remove the rough feeling of the woollen textile and to make the surface of the wool look smooth, not pilling. The woollen textiles can be finished by means of either wet or dry finishing. In yarn dyed fabrics, mere steaming may be enough; generally, however, the woollen textiles are washed and stentered (wet fixing) after weaving. In this connection, the finish of woollen textiles refers to wet finishing, which can also include dyeing treatment.

The term “woollen textile” in connection with this invention refers to wool fibre or wool fibre-containing tops, yarn, knitted or woven fabric containing at least 30%, preferably at least 50%, most preferably at least 70% of wool fibre. The wool fibre refers to fibre containing 100% wool. The wool tops or bay-yarn, in turn, can be pure wool, or the wool fibre may have been woven into or mixed with a synthetic fibre, such as polyacryl or polyester. The wool fibre may also have been woven into or mixed with some other protein-containing fibre, such as silk, or with some other

natural fibre, such as cotton or its derivatives, such as viscose. The tops refer to a bundle of fibres, from which the yarn is spun. The yarn is used to make knitted or woven fabrics and the finished textile products are then sown from them.

Generally, the reason for adding synthetic fibres to wool fibres in knitted and woven 5 fabrics is that the synthetic fibres are normally less expensive, whereby the price of the knitted or woven woollen fabric becomes more advantageous. Another reason is that the synthetic fibre makes wool-containing products dry quicker, which is of importance when making sports wear in particular. Synthetic fibres are added to knitted and woven woollen fabrics also because they enhance the strength of the 10 knitted or woven fabric.

In the method of the present invention, the properties of the wool fibre are influenced in particular. If the portion of wool fibre is less than 30%, the properties of the other fibres become dominant. For example, polyacryl and polyester pill fairly easily, and this property cannot be influenced by the present wool treatment 15 method. The larger the portion of wool in the woollen textile, the greater the impact of the method according to the invention on the properties of the woollen textile. Thus, the present method is the most advantageous when treating woollen textiles containing more than 70%, preferably more than 80%, and most preferably 100% of wool.

20 Enzyme treatment causes a decrease in weight of woollen textiles, and if the treatment is too strong, also in the strength of the textile. On the other hand, the decrease in weight of the textile indicates that the enzyme has worked. We measured the decrease in weight of the materials treated according to this invention, which decrease took place during the treatment and during the water washes following the treatments 25 (5 times the wool washing program). The measurement was carried out by means of weighing the original pieces of material, which were to be treated, before the treatment and both after the treatment and the following combined mechanical stage and final drying stage. The decrease in weight during the water washes was defined by weighing the materials before and after the washings, respectively, and 30 after this, the decrease in weight was measured in percentages. It was observed that the decrease in weight in the protease treatment was in a range of 9...13%. In washings after the treatments, further 9...20% decrease in weight took place.

On the basis of the weight loss measurements, it was established that the most advantageous conditions were provided by dosages of 0.025 ml/g to 0.05 ml/g. Under 35 these conditions, the weight loss in the protease treatment was 9 to 9.5% both in the protease treatments and the washings following the treatment. On larger dosages of

0.1 to 0.2 ml/g, the weight loss in the treatments was at a level of 12 to 13%, and in water washes following the treatments further 10 to 20% of weight loss took place.

Water wash of wool in connection with the invention refers to washing by means of a normal wool-washing program (the standard EN 60456) at a temperature of 40°C, 5 the program taking 45 to 70 min. The detergent used is a wool-washing agent; softeners can also be used.

As described above, excessively strong enzyme treatment may reduce the strength of woollen textiles, and too powerful mechanics combined with high temperatures may cause felting and dimensional changes. Furthermore, the temperature, at which 10 the mechanical drying was carried out, and the residual moisture contents achieved by continuing the mechanical drying seemed to have considerable effects. If drying was continued for too long, weakening of the strength of the woollen textile followed, whereas, if drying was stopped too early, the woollen textile, as a consequence, felted or interlaced in use and washings.

15 The weakening of strength of the woollen textiles was monitored by strength analyses. The strength analyses of woven fabrics were defined by means of a tensile testing machine method in accordance with the standard SFS 3981 (SIS 251231). The strength properties of the knitted fabrics were defined by measuring the abrasion resistance of the fabrics in accordance with the Martindale method SFS 4328 (BS 20 5690:1979).

On the basis of the strength analyses results, the strength loss of the woollen material that was treated with dosages of 0.1 ml/g or greater was over 50% compared 25 with a corresponding untreated woollen material. The instructions of the manufacturer of protease for the dosage were 0.125 ml/g at a minimum. On test dosages of 0.1 ml/g, the strength loss even at the most advantageous residual moisture levels was 53%. On dosages of 0.2 ml/g, the strength loss at the most advantageous residual moisture levels was from 56 to 60%. On a dosage of 0.05 ml/g and with residual moisture of 15 to 30%, the strength loss was 20%. On a dosage of 0.025 ml/g and with residual moisture of 10 to 30%, the strength loss was 14%. In practice, a 30 strength loss down to a level of 14% has not yet been found to have weakening effects on the textile. The best results of this test series were obtained with a dosage of 0.025 ml/g, when the residual moisture was 10 to 30%. Naturally, also on dosages of 0 ml/g, i.e., the reference samples, the strength losses were minor but, on the other hand, the measured working characteristics (the appearance and touch, and the 35 tendency to pill ) of the references did not improve either.

Strength analyses were also carried out after the wet washes. At the most advantageous dosage and residual moisture levels, the strength was decreased from 10 to 14% during the water washes.

5 The abrasion resistance was defined by the Martindale method in accordance with the standard SFS 4328 (BS 5690:1979). The abrasion resistance measures the wear and tear properties of the woollen textile. The abrasion resistance result indicates the number of rotations needed for the textile to wear out upon rubbing the woollen textile under examination against a friction surface. The abrasion resistance is assessed as the number of rotations needed for the wearing surface of the textile to 10 break.

15 "Lint forming or pilling" refers to the small fibre bundles or lint, which are formed on the surface of the woollen textile. The tendency to pill was defined by rubbing the samples by means of the Martindale method for 125, 500 and 2000 rotations, after which the rubbed samples were evaluated by means of a scale of reference in accordance with the standard SFS 3378 on the basis of the appearance of the samples. The value 5 on a scale of 0 to 5 signifies the smallest amount of pilling. The term "no substantial lint" means that the majority of the textile surface is free of 20 lint, i.e., the pilling value is in a range of 3 to 5.

25 In connection with the invention, it was discovered that the tendency to form lint decreases directly in proportion to increasing the dosage. On dosages of 0.1 ml/g and higher, the pilling values at the most advantageous residual moisture contents for all revolutions of rubbing (125, 500 and 2000) were in a range of 4.0 to 4.7. Under the most advantageous conditions from the point of view of both the dosages and the residual moisture contents, the pilling values on dosages of 0.025 ml/g were in a range of 3.5 to 4.2 and on dosages of 0.05 ml/g in a range of 3.5 to 4.5. The pilling values of the most advantageous finishing conditions correspond to the common quality requirements of those who buy products made of woollen materials, which is at the level of 3 to 4.

30 The woollen textiles that were treated under the conditions of protease treatment, which were the most advantageous to the woollen textiles (the protease dosage was 0.0125 ml/g and the residual moisture content in the mechanical drying stage was from 10 to 30%), lost a maximum of 14% of their strength in the treatments, and shrank in the washings by 0.7% at a maximum in the direction of the warp, and by 0.8% at the maximum in the direction of the weft. The pilling values of these 35 samples were in a range of 3.5 to 4.2%, the touch had softened in the treatments the most and, in addition, the softened touch kept well in the washings.

The term "felting" or "interlacing" refers to the effect on the appearance of woollen textiles caused by shrinkage.

The term "shrinkage" means that the size of the woollen textile has decreased, i.e., a dimensional change has taken place. This is indicated as percent of the size of the untreated material. It is given separately in the direction of the weft and the warp for both woven and knitted fabrics. Some shrinkage takes place during the finishing of the woollen textile; however, this is of no importance to the consumer. The shrinkage that takes place after finishing is essential. If the shrinkage after finishing is less than 3%, it is acceptable. Shrinkage greater than this affects the appearance of the textile, making the textile look felted. This invention is concerned with monitoring the shrinkage, which takes place during finishing and which generally varied from about 3% to 5%. The shrinkage that took place after finishing was measured after 5 times of washing. In this invention, the amount of shrinkage was less than 3% after finishing and, generally, varied within 0% and 2%. The term "no substantial felting" means that the textile has not shown essential shrinkage, i.e., the shrinkage is less than 3%.

The dimensional changes were defined in accordance with the standard SFS 5157 (ISO 5077-1984). For the definition of the dimensional changes, areas of 50 cm x 50 cm were marked on the pieces of material, enabling measurements in the direction of the weft and the warp, which had taken place in finishing and in the subsequent water washes.

The dimensional change that took place during the finishing (the 1<sup>st</sup> dimensional change) was measured and given in % of the original untreated material. The dimensional change that took place in 5 water washes (the 2<sup>nd</sup> dimensional change) was defined and given in % of unwashed and finished material. The shrinkage of the woollen material that took place during the finishing (the 1<sup>st</sup> dimensional change) in the direction of the warp was in a range of 1.8 to 4.5% and in the direction of the weft in a range of 0 to 5%. Under the most preferable conditions, it was 2.8 to 4% in the direction of the warp, and 0.5 to 2.5% in the direction of the weft. After five water washes, the dimensional change of the finished materials in the test series varied in the direction of the warp in a range of 0 to 1.9% and in the direction of the weft from 0 to 4.9%. The dimensional change of the samples that were finished under the most advantageous conditions after the water washes was 0 to 1% in the direction of the warp, and 0.2 to 0.8% in the direction of the weft. The dimensional change of the original material after five water washes was 4.5% in the direction of the weft and 5.0% in the direction of the warp.

The appearance and the touch were evaluated by panel determinations. The panel consisted of five judges, and the results given by them were used to calculate a mean value, which was then given as the result. The appearance and the touch of the materials were graded on a scale of 5, 4, 3, 2 or 1 minuses, 0 or 1, 2, 3, 4 or 5 plusses. The touch was evaluated for the softness of the materials. The appearance was visually evaluated for a trim and smooth surface of the material, for the opening of the stitches and the overturning and bias of the stitches and the wales. When giving the grades, an original unfinished woollen woven or knitted fabric was used as a reference.

- 5 10 According to the touch and appearance grading, all samples that were treated felt softer than the original untreated samples. The references were the ones that showed the least degree of softening among the treated samples. The samples, wherein the residual moisture content after mechanical drying stage was 15 to 30%, had the softest touch.
- 15 20 The method according to the present invention can be used to treat wool fibre, tops, yarn, knitted fabric, woven fabric or finished articles made of the knitted or the woven fabric. The process can just as well be implemented for metre material and ready made products. The materials containing the above-mentioned wool fibre are herein called woollen textiles.
- 25 30 In the finishing method of woollen textiles according to the invention, a woollen textile in an aqueous solution is brought into contact with a protease enzyme in a large amount of water so that the woollen textile is moved as little as possible or not at all at a temperature of 60°C or less. Taking into consideration any errors in the equipment and methods, the temperature of about 60°C herein also refers to temperatures that are 1 to 3° higher or lower.

The treatment time can be from 10 to 90 min. For woven fabrics, the treatment time is preferably 15 to 60 min, more preferably 15 to 30 min. For knitted fabrics, the treatment time is preferably 15 to 60 min, more preferably 15 to 45 min.

- 30 The protease enzyme is preferably an alkaline protease, most preferably serine protease. Proteases of various manufacturers are suitable for the purpose, however, preferably proteases that the manufacturers recommend for the treatment of wool. Such proteases include, e.g., Genencor's Multiplus L or Gentle L, Novo's Novolan L or Novon Savinase.

- 35 With this invention, it was established that it is preferable to carry out the protease treatment under neutral or alkaline conditions. The pH is preferably adjusted to a range of 6 to 11, more preferably to a range of 7 to 11, particularly to a range of 7 to

9 or even 9 to 11. Of course, the pH the manufacturer recommends for the proteases used should be taken into account. For example, the manufacturer recommends that Genencor's Protex Multiplus L enzyme preparation should be used at pH of 7 to 9.5, Protex Gentle L at pH of 6.5 to 10, Novo's Novolan L at pH of about 8.5, 5 Novo's Savinase at pH of 8 to 8.5. In connection with the invention, it was observed that the appearance and the touch of woollen textiles even improved some, when the pH was raised to 11, while the temperature was 50°C.

With this invention, it was observed that, under alkaline or neutral conditions, wool endures a temperature of about 60°C. On the other hand, it is even more advantageous to have a temperature of less than 60°C, typically, 35 to 55, preferably 40 to 10 60°C, more preferably 40 to 50°C, most preferably about 50°C (the temperature of about 50°C herein also refers to any temperatures that are from 1 to 3°C lower or higher). On the other hand, in the protease treatment, the temperature optimum of the enzyme used should also be taken into account. In connection with the invention, 15 it was discovered that at 50°C, the activity of the Protex Multiplus L enzyme at pH 11 was as good as at pH 9, but at 60°C, the activity in the corresponding acid contents was lower.

As stated above, it was observed in connection with the invention that the amounts of protease recommended by the enzyme manufacturers had a weakening effect on 20 the abrasion resistance of woollen textiles. A desired effect was achieved using smaller dosages than those recommended. For example, when using Genencor's Protex Multiplus L protease product, which according to the dosage instructions should be dosed in an amount of 0.125 to 1 ml/g of dry woollen textile, good results 25 were obtained with a dosage as low as 0.0125 ml/g, and the strength was found to deteriorate with dosages of more than 0.1 ml/g. Hence, it is preferable to use amounts of enzyme that are on the lower level of the recommended amounts of enzyme. Calculated in proteins, 0.1 ml/g corresponds to 3.5 mg/g. According to a preferred embodiment of the invention, it is preferable to dose the enzyme preparations, as calculated in proteins, in amounts of about 0.4 to 4.4 mg/g of dry woollen 30 textile.

In conclusion, the dosage for knitted fabrics is preferably less than 8 mg/g of dry textile, more preferably less than 4.4 mg/g; most preferably less than 3.5 mg/g of dry textile. For woven fabrics, which are tight textile structures, larger dosages and greater amounts of mechanics can be used than for the knitted fabrics, which have 35 slacker structures. A preferred dosage for woven fabrics is less than 35 mg/g of dry textile, more preferably less than 17.5 mg/g, most preferably less than 8 mg/g of dry textile.

In addition to the enzyme dosage, increasing the amount of mechanics and lengthening the enzyme treatment time also had a weakening effect on the strength of the woollen textiles.

5 In connection with the invention, it was observed that, in the enzyme treatment, it is preferable to use as little mechanics as possible. As the examples indicate, three different levels of mechanics 0, 1 and 2 were used in an open drum machine in the wet processes of the invention. Mechanics 0 corresponded to a rotation speed of 4.0 rpm, mechanics 1 corresponded to 6.0 rpm and mechanics 2 corresponded to 10.0 rpm. The rotation speeds of 4 to 6 rpm weakened the strength the least. When using 10 other machines, it is preferable to select the rotation speeds from the corresponding range.

In the method according to the present invention, wool fibre in an aqueous solution is brought into contact with a protease enzyme in a large amount of water. The large amount of water in this connection refers to the fact that the ratio of the weight of 15 the knitted or woven fabric under treatment to the weight of the water is at least 1/10; the liquor ratio is preferably in a range of 1/20 to 1/40, the liquor ratio is most preferably about 1/30.

After the enzyme treatment, it is preferable to inactivate the enzyme without raising the temperature to above 60°C (to take into account any errors of the equipment and 20 the methods, the temperature of about 60°C herein also refers to any temperatures that are 1 to 3°C higher or lower). This is the case in particular, when the conditions in the treatment solution are neutral or alkaline. In that case, it is not preferable to inactivate the enzyme by means of temperature (i.e., increasing the temperature). Recommendable inactivation methods of enzyme include, e.g., chemical inactivation, 25 such as lowering the pH sufficiently low, to acidity of pH 4 to 5, or by adding copper ions, for example. On the other hand, when the pH is kept low enough, woollen textiles seem to endure fairly high temperatures. The pH can be lowered to less than 5, preferably to a range of 4 to 5 or below pH 4, but, in practice, it is difficult to adjust the pH below 4. At a pH of less than 5, wool may endure temperatures 30 as high as over 90°C without felting. If dyeing treatment is carried out after the enzyme treatment, the enzyme does not need to be separately inactivated, as the enzyme is inactivated under the dyeing conditions. To inactivate the enzyme at below pH 5, a treatment time of 5 to 15 min, preferably 5 to 10 min is sufficient.

Inactivation of protease enzymes by means of copper ions is described in the publication JP 2001262474.

After inactivating the enzyme, before the mechanical treatment stage, moisture should be removed from the woollen textile so as to obtain a moisture content of about 50 to 70%. This can be carried out by centrifugation. After this, the woollen textile is taken to drying by a mechanical drying method, such as drum drying or 5 tunnel drying. Naturally, drying is quicker at a high temperature, but the temperature of the mechanical stage in this method must not increase to above about 60°C, preferably not to above about 50°C. It is preferable to treat woollen textiles in the mechanical stage so as to achieve a certain final moisture content, which is 10 to 10 45%, preferably 10 to 30%. For the equipment used in the examples of the invention, this means that the drum or tunnel drying times are short, less than 10 min, preferably 5 to 10 min, most preferably about 6 min (+/- 1 min). With this invention, it has been observed that it is preferable to make the woollen textile dry by 15 means of a method, wherein it is subjected to a suitable amount of mechanical action; otherwise, the woollen textile is felted or interlaced later on. If the woollen textile is dried mechanically for too long, its strength decreases.

Thus, the residual moisture content is 35 to 40% at a maximum, 5 to 10% at a minimum, and preferably 10 to 20%.

It is preferable to allow the final drying of the product to take place without mechanics, for example, flat or in a hanging form. This is most preferably carried out 20 at room temperature, which refers to a temperature of about 18 to 30°C, most commonly about 20 to 25°C.

The dyed woollen textiles can be treated with enzyme or the dyeing treatment can be carried out after the enzyme treatment. In this invention, it was established that the latter, in particular, brings about extremely good dyeing results. Dyeing can be 25 carried out by means of the methods well known by those skilled in the art. In connection with the dyeing, the temperature increases to over 90°C; typically, the temperature is 90 to 98°C, preferably 90 to 95°C, but when the pH is below 5, typically in a range of pH 4 to 5, the wool withstands the dyeing well. In connection with the invention, it was established that it is advantageous to carry out the enzyme treatment and the dyeing treatment in the same wet process without a drying stage between the treatments. As the enzyme does not work under the dyeing conditions, the effect of the enzyme is terminated or it stops before the dyeing stage. During dyeing, it is preferable to use a somewhat higher degree of mechanics than during the protease treatment, otherwise, the result will be uneven. For the equipment used in 30 this invention, it was preferable to adjust the mechanics to 6 - 10 rpm during dyeing.

When dyeing wool, any commonly used and commercially available wool dyestuffs can be used. Dyeing can be carried out at temperatures of about 90 to 98°C, preferably at temperatures of 90 to 95°C, with a pH of 4 to 5. Under these conditions, the wool withstands treatments of as much as about 1 hour in duration. When dyeing 5 wool, so called reactive dyestuffs can also be used, whereby temperatures as high as the above and a low pH are used, but part of the treatment is carried out at lower temperatures of about 40 to 60°C.

The most advantageous conditions for the protease treatment are the same for the 10 same material independent of whether the material is yarn dyed or if the material is dyed in the same wet process with the protease treatment.

According to the preferred embodiments of the present invention, it is important that the wool fibre is not subjected to a temperature of over about 60°C in any other stage except during dyeing. If the temperature is raised to over 60°C, when inactivating the enzyme, for example, the pH should be low, i.e., in a range of 4 to 5 or 15 less. Another essential fact is that, under no circumstances should the wool fibre be subjected to excessive mechanical stress, especially not during the wet process. It is also of importance to leave a suitable residual moisture content to the woollen textile after the mechanical treatment and before final drying, which is preferably carried out without mechanics. Neither the amounts of enzyme should be so great so as 20 to essentially weaken the woollen textile. The method according to the present invention is a combination of the stages of a method, where in every step, a wrong type of stress on the wool fibre is avoided. By using the finishing method according to the preferred embodiments of the present invention, woollen textiles can be manufactured, which last at least 5 times washing with water according to the wool 25 instructions. Indeed, it has been observed that the woollen textiles that were treated with the method according to the most advantageous embodiments of the invention withstood well as many as 10 or 20 washings without shrinking more than 3%, and without essentially felting or pilling or becoming rougher in the water wash. It was also established, that one washing after the mechanical treatment further improved 30 the touch and the appearance of the wool textile. This effect was most obvious in wool wash that is preferably carried out with cool water at about 30°C.

In the method according to the preferred embodiments of the invention, it is essential that the softness and pilling of the woollen textiles are controlled by means of a correct enzyme treatment, and the dimensional change of the textile is controlled by 35 means of a correct process.

## Examples

The tests of the examples employed the following machines and equipment: The protease and dyeing treatments were carried out in the open drum machine Wasca-tor FOM 71 Special. The diameter of the inner drum was 515 mm, the depth 335 mm, and the volume 70 dm<sup>3</sup>. The drum drying stage was carried out by means of the drying and punching machine LAKO KA 901, which includes an ITARA air-circulation system. The inner diameter of the drum was 1600 mm, the length 1350 mm, the volume 2700 l, and the rotational speed 30 rpm. The final drying stage was carried out in a hanging form in a steam tunnel VEIT (tailor made system) using hot-air chambers only, in other words, the steam in the steam chambers was not on. The tunnel contains 1 steam chamber and 3 air chambers. In these tests, only the air chambers were used, which comprise continuous heat regulation and had been adjusted to room temperature, 24°C.

In the examples, both woven and knitted woollen fabrics were treated, which had been made from 100% fine Merino wool yarn, which was spun by the worsted method and had a thickness of NM 2/28. The yarn was off-white and it had been treated to be ready for dyeing, i.e., washed and steamed after spinning.

The woollen textiles had been cut into pieces of 60 cm x 60 cm for the tests.

After the finishes, mechanical testing was conducted on the treated woollen textiles by means of standard methods. Regarding the materials treated, the weight loss in the treatment and the water washes (5 x wool wash program) after the treatments were measured. It was carried out by weighing the original pieces of material to be treated before and after the treatment and after the following combined drum and tunnel drying. The weight loss in water washes was defined by weighing the materials before and after the washes, respectively, and after this, the weight loss was calculated in percentage.

The dimensional changes were defined in accordance with the standard SFS 5157 (ISO 5077-1984). For the dimensional change definitions, areas of 50 cm x 50 cm had been marked on the pieces of material for measuring the dimensional changes in the direction of the weft and the warp, which had taken place in the finishes and the subsequent water washes.

The strength measurements of the woven fabrics were defined by means of the tensile testing machine method in accordance with the standard SFS 3981 (SIS 251231).

The abrasion resistance was defined by means of the Martindale method in accordance with the standard SFS 4328 (BS 5690:1979).

The pilling tendency was defined by rubbing the samples by the Martindale method for 125, 500 and 2000 rubs, after which the abraded samples were assessed by means of a comparison scale according to the standard SFS 3378 on the basis of the appearance of the samples. On a scale of 0 to 5, the value 5 refers to the smallest amount of pilling.

5 The assessments for appearance and touch were carried out by panel determinations. The panel consisted of five judges, whose results were used to calculate a mean value, which was then used as a result. The appearance and the touch of the materials were assessed on a scale of 5, 4, 3, 2 or 1 minuses, 0 or 1, 2, 3, 4 or 5 plusses. In the assessment of the touch, the softness of the materials was assessed. 10 The assessment of the appearance was used to visually assess the trim and the smoothness of the surface of the material, the opening of the stitches and the overturning and the bias of the stitches and the wales. When giving the values, original unfinished woven or knitted woollen fabrics were used as references.

15 **Example 1**

The woollen fabric used was woven from the yarn mentioned above; the weave was 1 x 1 plain weave, the basis weight 190 g/m<sup>2</sup>. A series of 21 trial samples was conducted. The size of a trial sample was 1716 g.

20 In the tests, Genencor's protease enzyme (Genencor Protex Multiplus L) was used. Serine proteases of also other manufacturers had been used in preliminary tests, but no significant differences were observed between them.

25 The amount of protease dosed was 0 or 0.025 or 0.05 or 0.1 or 0.2 ml/g of dry woollen textile. 0.1 ml of Genencor's Protex Multiplus L protease corresponds to 3.5 mg of protein. Other proteases could be dosed, correspondingly, calculated as protein grams per dry woollen textile.

30 The manufacturer of protease recommends an amount of 0.125 to 1 g/l. In all the test series of the examples, the dosages recommended by the protease manufacturers were tested, which dosages were found to be detrimental to the strength properties of the woollen textiles. The treatments were carried out in a liquor ratio of 1:30, which in the preliminary tests was found to be large enough. The manufacturer of the enzyme had recommended a pH between 7 and 9.5, so the treatment in these tests was conducted at pH 9. When repeating the tests at pH 11, the touch and the appearance of the woollen textile were discovered to be as good as or better than at pH 9. The temperature in the protease treatment was 50°C. The treatment time was 35 30 min. The treatment was carried out in the above-mentioned open drum machine (machine 1). The mechanics of the machine were adjusted to the value 2.

In the wet treatments of the tests, three different levels of mechanics: 0, 1 and 2 were used. The mechanics 0 corresponded to a rotational speed of 4.0 rpm, the mechanics 1 corresponded to 6.0 rpm and the mechanics 2 corresponded to 10.0 rpm.

5 The protease treatment was ended by the inactivation of the enzyme at a temperature of 60°C. The pH was adjusted to a level of 4 for 15 minutes. After this, rinsing at a temperature of 30 to 40°C was carried out for 10 minutes. Then, the treated woollen materials were spin-dried so as to obtain a moisture content of 50 to 70%, which in this test corresponded to a spinning time of 2 minutes. Thereafter, the test material was brought to drying in tumble-drying (machine 2) at 50°C so as to obtain 10 certain residual moisture content of 5 to 35%. In this test series, 4 different levels of residual moisture contents were tested, being 5 to 10%, 10 to 15%, 15 to 30% and 30 to 45%.

15 Table 1 shows the circumstantial variables for this test series (the protease dosage and the residual moisture content), the weight loss in the protease treatment and in the water washes following the same. The weight loss of the woollen textiles during the finish and in the water washes after the finish was measured and given in % of the original weight of the untreated woollen textile. The weight loss was stated to be directly proportional to the efficiency of the protease treatment. With the dosage increasing, the weight loss increased, correspondingly.

20 According to the results, the weight loss in the protease treatments of the test series was in a range of 9 to 13%. The residual moisture content had no effect on the weight loss in the treatment or the subsequent washes. In the washes subsequent to the treatments, a further weight loss of 9 to 20% took place. On the basis of the weight loss measurements, the most advantageous conditions were the dosages of 25 0.025 to 0.05 ml/g in combination with the residual moisture contents of 10 to 30% after tumble-drying. The weight loss occurring under these conditions was 9 to 9.5% both in the protease treatments and the washes subsequent to the treatment. On larger dosages of 0.1 to 0.2 ml/g, the weight loss in the treatments was at a level of 12 to 13%, and in the water washes subsequent to the treatment, a further weight 30 loss of 10 to 20% took place.

The dimensional changes are shown in Table 2. The dimensional change that took place during the finish (the 1<sup>st</sup> dimensional change) was measured and given in % of the original untreated material. The dimensional change that took place in 5 washes (the 2<sup>nd</sup> dimensional change) was defined and given in % of the unwashed, finished 35 material. The shrinkage of the woollen material during the finish (the 1<sup>st</sup> dimensional change) in the direction of the warp was in a range of 1.8 to 4.5%, and in the direction of the weft in a range of 0 to 5%. In the most advantageous conditions

(samples 6, 7, 10 and 11), it was 2.8 to 4% in the direction of the warp and 0.5 to 2.5% in the direction of the weft. The dimensional change of the finished materials after five washes varied in the test series in the direction of the warp in a range of 0 to 1.9% and in the direction of the weft 0 to 4.9%. The dimensional change of the 5 samples finished in the most advantageous conditions (samples 6, 7, 10 and 11) after the washes was 0 to 1% in the direction of the warp and 0.2 to 0.8% in the direction of the weft. The dimensional change of the original material after five washes was 4.5% in the direction of the weft and 5.0% in the direction of the warp.

Table 3 shows the results of the strength and pilling measurements. On the basis of 10 the strength measurement results, the loss in strength of the woollen material treated with dosages of 0.1 ml/g or higher was over 50% compared with corresponding untreated woollen material. The instructions of the protease manufacturer for the dosage were 0.125 ml/g at a minimum. On the 0.1-ml/g dosages of the tests, the loss in strength even at the most advantageous residual moisture content levels was 53%. 15 On dosages of 0.2 ml/g, the loss in strength at the most advantageous levels of residual moisture contents was 56 to 60%. On a dosage of 0.05 ml/g and with a residual moisture content of 15 to 30%, the loss in strength was 20% (samples 10). On a dosage of 0.025 ml/g and with a residual moisture content of 10 to 30%, the loss in strength was 14% (samples 6 and 7). In practice, the loss in strength to the level of 20 14% has not yet been found to have any weakening effects on the textile. The best results of this test series were obtained with a dosage of 0.025 ml/g, when the residual moisture content was 10 to 30%. Naturally, also with dosages of 0 ml/g, i.e., the reference samples, the losses in strength were minor but, on the other hand, the working characteristic measured (the appearance and the touch and the tendency to 25 pill) did not improve either.

Strength measurements were also carried out after washing with water. At the most advantageous dosage and residual moisture content levels, the strength was reduced by 10 to 14% in water washes (samples 6, 7, 10 and 11).

The results of the pilling measurements are shown in Table 3. On the basis of the 30 results, the tendency to form lint decreases directly in proportion to the increase in the dosage. On dosages of 0.1 ml/g and higher, the pilling values in the most advantageous residual moisture contents on all cycles of abrasion (125, 500 and 2000) were in a range of 4.0 to 4.7. Under the most advantageous conditions, from the point of view of both the dosages and the residual moisture contents, the pilling 35 values on dosages of 0.025 ml/g were between 3.5 and 4.2, and on dosages of 0.05 ml/g, between 3.5 and 4.5. The pilling values of the most advantageous finishing

conditions correspond to the common quality requirements of the buyers of articles made of woollen materials, which are on a level of 3 to 4.

According to the assessments of the touch and the appearance, all samples that were treated felt softer than the original untreated samples. Regarding the treated samples, the references showed the least degree of softening. As regards its touch, sample 6 was the softest in the test series, i.e., it showed the highest degree of softening in the treatments. In addition, the soft touch of sample 6 was the best in the series to maintain its softness in washing. Sample 7 had the second best touch in the series, i.e., it showed the second highest degree of softening in the treatments, the touch having also survived the washings.

The samples 4, 8, 12, 16 and 20, which had the maximum residual moisture content after the mechanical drying stage, felt only a little softer than the reference samples, and so did the samples 1, 5, 9, 13 and 17, which had a corresponding minimum residual moisture content.

On the basis of the results of the Example 1 test series, it could be concluded that the mechanical drying stage (the tumble-drying stage) is necessary for improving the working characteristics. The residual moisture content, which remains in the woollen material after the mechanical drying stage, is also a circumstantial variable of great importance on the basis of this test.

The woven woollen fabrics, which were treated under the conditions of the protease treatment that are the most advantageous for the woollen fabric of the example (a protease dosage of 0.0125 ml/g and a residual moisture content of 10 to 30% after the mechanical drying stage), lost a maximum of 14% of their strength in the treatments, and shrank a maximum of 0.7% in the direction of the warp, and a maximum of 0.8% in the direction of the weft in the washings. The pilling values of these samples were between 3.5 and 4.2, the touch had softened the most in the treatments and, in addition, the softened touch had endured the washings well.

### **Example 2**

The above-mentioned woollen yarn was dyed and 1/1-rib knit, i.e., plain fabric, was made of the dyed yarn for the protease treatment testing of dyed knitted fabrics. The basis weight of the knitted fabric was 375 g/m<sup>2</sup>.

A series of 10 trial samples was conducted to find a level of dosing and mechanics of protease treatment that would be suitable for knitted materials. The size of the trial sample was 600 g. The enzyme of Example 1 was used. The numbers of the samples of the dyed materials of the test series (dyestuff 1) were 30 to 39. On the basis of these test results, the protease treatment testing of dyed knitted fabric was

continued using a test series of 20 tests, wherein the numbers of the samples of the dyed material (dyestuff 2) were 41 to 61.

5 The circumstantial variables in the tests comprised the protease dosage, the protease treatment time, the pH of the treatment, and the level of mechanics. The amounts of the protease dosed were 0 or 0.0125 or 0.025 or 0.125 or 0.250 ml/g.

10 The treatments were carried out in a liquor ratio of 1:30, which in the preliminary tests was found to be large enough also for knitted fabrics. The manufacturer of the enzyme had recommended a pH of between 7 and 9.5, so the treatment in these tests was carried out at the pH values of 7 and 9.5. The treatment times were 15 min and 10 min. The temperature in the enzyme treatment was 50°C. The treatment was carried out in an open drum machine (machine 1). The mechanics of the machine was 0 or 1 or 2.

15 Inactivating the enzyme at a temperature of 60°C terminated the enzyme treatment. The pH was adjusted to the level of 4 for 15 minutes. After this, rinsing was carried out at 30 to 40 degrees for 5 minutes. Then the woollen textile was spin-dried so as to obtain a residual moisture content of 50 to 70%. This corresponded to a centrifugation of 2 minutes. After this, the test material was taken to dry in tumble-drying (machine 2) at 50°C so as to reduce the residual moisture content to a level of 10 to 20 30%. On the basis of the preliminary tests, this residual moisture content had been found to be the most suitable. Tables 5, 6 and 7 show the circumstantial variables of the dyestuff 1 of the test series (the sample numbers of 30 to 39) and the results of the measurements. Tables 8, 9 and 10 show the circumstantial variables of the dyestuff 2 of the test series (the sample numbers of 40 to 61) and the results of the measurements.

25 Table 7 shows the assessments for the appearance and the touch of dyestuff 1. On the basis of the test series of the dyestuff 1, the level of mechanics 2 was too strong, causing, according to the touch and appearance assessment, felting or interlacing as early as in the finishing treatments (samples 35, 37 and 39). The felting of the materials in the treatments corresponded to a shrinkage of over 10% in the dimensional 30 changes.

35 Regarding its appearance and touch, sample 32 was the best after the treatment and, in addition, these properties had endured the washings best. Sample 38 was assessed to have an equal touch after the treatment, but its appearance was slightly better than that of sample 36. In samples 36 and 38, the stitch pattern had slightly opened in the treatments and the stitches showed a slight bias, making the surface of the knit more uneven compared with the untreated knitted fabric.

According to the assessments of the appearance and the touch and regarding the circumstantial variables of the protease treatments, pH 9.5 was more advantageous than 7, and the mechanics 0 was more advantageous than the mechanics 1.

Shortening the treatment time from 30 minutes to 15 minutes and, at the same time, 5 increasing the dosage to 10-fold (Sample 34) or to 20-fold (Sample 33) did not provide better results.

The dimensional changes of the test series of dyestuff 1 were defined as in Example 1. The 1<sup>st</sup> dimensional change and the 2<sup>nd</sup> dimensional change are shown in Tables 5. The felting of the materials in the treatments is shown as shrinkages of over 10%.

10 In the materials of mechanics 0 and 1, which had not felted, the 1<sup>st</sup> dimensional changes in the direction of the warp were between 3.3 and 9.3%, and in the direction of the weft between -3.1 and 1.0%. For the materials that had been graded high in the assessments for touch and appearance, the 2<sup>nd</sup> dimensional changes in the direction of the warp were between -0.2 and 1.0%, and in the direction of the weft 15 between -1.0 and 0.7%. After water washes, the unfinished materials (samples 30) shrank by 5.1% in the direction of the warp, and stretched by 4.2% in the direction of the weft.

The pilling and abrasion resistance results are shown in Table 6. These properties were not measured from the materials that felted in the treatments. The pilling values 20 of the samples that were graded high in the appearance and touch assessments (32, 36 and 38) were between 3.0 and 4.5, whereas the corresponding values for the unfinished samples were between 1.5 and 3.0. The abrasion resistance of the samples 32, 36 and 38 decreased 11.6 to 14.2% compared with the abrasion resistance value of the unfinished material. The pilling values of the samples 33 and 34, which 25 were not graded high in the appearance and touch assessments but had not felted in the treatments or washings either, were in a range of 2.0 to 4, and the loss in abrasion resistance in the treatments was from 16 to 24% compared with the corresponding value of the original unfinished sample.

On the basis of the results, it was concluded that the materials, which were graded 30 high in the appearance and touch assessments after the finishes, also showed minor dimensional changes in washings. In addition, the pilling values of these materials were at the level of the common quality requirements. Neither did the abrasion resistances of these samples diminish more than 14% in the treatments.

On the basis of the test results of dyestuff 1, the mechanics levels 0 and 1 were selected 35 for the testing of dyestuff 2. The other variables selected were treatment

times of 15 and 30 min and pH levels of 7 and 9.5. The dosages were 0 or 0.0125 or 0.125 ml/g.

The touch and appearance assessments of dyestuff 2 are shown in Table 10.

5 Sample 58 was graded the highest in the touch and appearance assessments of this test series, its touch having become soft and pleasant, still retaining its woolly nature. The woolly touch also remained in washings.

The touch of sample 47 (pH 7) after the treatment was as good as that of sample 58 (pH 9.5) but the appearance was somewhat more uneven.

10 The appearance of sample 50 (mechanics 0) after the treatment was better than that of sample 47 (mechanics 1), and the better appearance of sample 50 also kept well in the washings.

Sample 59 (mechanics 1) was more uneven than sample 58 (mechanics 0) after the treatment.

15 The appearance of sample 60 (dosage 0.125 ml/g, the mechanics level 0) after the treatment was as good as sample 58, i.e., trim, but the touch had turned viscous. The viscous touch remained unchanged in the washings.

20 The appearance of sample 61 (dosage 0.125 ml/g, mechanics 1) was trim after the treatment, but the touch had turned viscous. The viscous touch had increased in washings. The samples, which had been graded high for the appearance and the touch, had in the protease treatment a pH of 7 and the mechanics level 0.

Regarding the samples, which, in addition to the above-mentioned levels of pH and mechanics, had a dosage of 0.0125 ml/g, the woolly touch as well as the good and trim appearance after the treatment remained unchanged in the washings.

25 Table 8 shows the dimensional changes of the test series, which were defined as in the previous tests. The dimensional changes of the samples (the 2<sup>nd</sup> dimensional changes), whose appearance and touch were graded high, were in washing between -1.0 and 1.2% in the direction of the warp, and between -0.6 and 0.3% in the direction of the weft. Both the unfinished materials (samples 52) and the references (samples 44, 45, 46 and 51) had experienced dimensional changes of over 3% in the 30 water washes.

The dimensional stability of other than the samples that were graded high in the touch and appearance assessments, and that of the unfinished samples and the references was in a range of -2.1 to 2.9% in the water washes.

The abrasion resistance and the pilling results of dyestuff 2 are shown in Table 9. The abrasion resistance had decreased in proportion to the dosages so that the greater the dosage, the more the abrasion resistance had decreased. When the mechanics of the treatment was increased, the abrasion resistance correspondingly further decreased, the dosages of the protease treatment being the same. Increasing the time under the same conditions also had a weakening impact on the abrasion resistance. The changes in the abrasion resistance in the finishes of the samples, which were given plusses in the appearance and touch assessments, were between 8 and 20%. The abrasion resistance of the reference had decreased by less than 1%.

5 The abrasion resistance of the samples, which had a dosage of 0.0125 ml/g in the protease treatment, had decreased by 5.5 to 12% in the treatments. The samples with a protease treatment dosage of 0.125 ml/g showed a decrease of 14 to 21.3% in the abrasion resistance.

10 The abrasion resistance of the sample (58), which in the appearance and touch assessments was graded the highest, was reduced by 10.7%.

15 The abrasion resistance of the sample (58), which in the appearance and touch assessments was graded the highest, was reduced by 10.7%.

15 On the basis of the pilling results, the pilling results of the samples, which in the appearance and touch assessments had gained at least 2 plusses, were between 3 and 5. The pilling values of the samples that had gained 1 plus varied in a range of 2.5 to 4, and those of the references in a range of 1.5 to 3. The pilling values of the untreated samples remained in a range of 1 to 3.

20

### Example 3

In these tests, the off-white knitted woollen fabric that was described at the beginning of the examples, comprising 1/1 ribbing and having a basis weight of 430 g/m<sup>2</sup>, was used, and subjected to the combined protease and dyeing treatments.

25 A series of 16 tests was conducted. The size of the trial sample was 300 g. The circumstantial variables comprised the dyestuff, the dosage and pH of the protease treatment, the time and the mechanics. The amount of protease dosed was 0 or 0.0125 or 0.125 or 0.25 ml/g. The pH was 7 or 9.5. The mechanics was 0 or 1.

The protease treatments were conducted either before or after the dyeing treatment.

30 The protease treatments were carried out in a liquor ratio of 1:30. The pH was 7 or 9.5. The treatment time at pH 7 was 15 min and at pH 9.5 30 min. The temperature in the enzyme treatment was 50°C. The treatment was carried out in the above-mentioned open drum machine (machine 1). The mechanics of the machine was 0 or 1.

The enzyme treatment was terminated by inactivating the enzyme at a temperature of 60°C by adjusting the pH to a level of 4 for 15 minutes. After this, rinsing was carried out for 5 minutes at 30 to 40°C. Thereafter, the liquor was changed, followed by the dyeing stage using a normal woollen textile dyeing method at a pH 5 level of 4 to 5 and at a temperature of 90°C, the fixing time varying between 20 and 30 minutes, depending on the shade. After this, rinsing was carried out at 40°C for 5 minutes. Then, the procedure was continued by the centrifugation and drying methods according to the previous protease treatments. In this way, the so-called enzyme and dyeing tests were carried out, the samples 64, 66, 77, 78, 79, 80 and 81.

10 In the so-called dyeing and enzyme tests 62, 63 and 65, first, the above-mentioned dyeing treatment was carried out, followed by the protease treatment so as to cool down the treatment liquor to 50°C after the dyeing and to exchange the liquor for a new one, wherein the protease treatment, which was described in connection with the previous tests, with its spinning and drying stages was carried out.

15 Furthermore, so-called enzyme, drying and dyeing tests were carried out, the samples 67, 68, 69 and 70. First, the protease treatment was carried out, as described in the first tests of this series. After this, the spinning and the tumble-drying stage of the drying method were carried out so as to obtain a residual moisture content of 10 to 35% of the woollen textile. Thereafter, the dyeing treatment was carried out as 20 described in the first tests of this series. After the dyeing, spinning and drying were carried out, as after the protease treatments of the previous tests.

The dimensional changes were defined as in the previous tests and the results are shown in Table 10. The dimensional changes subsequent to the washings, i.e., the 2<sup>nd</sup> dimensional changes for the references (materials treated with a buffer and dyed) were in the direction of the warp between 3.7 and 4.5% and in the direction of the weft between -3.6 and 4.2%. The dimensional changes subsequent to the washings were minor for the samples dyed by the protease treatments, i.e., they were in the direction of the warp in a range of 0.6 to 1.8% and in the direction of the weft in a range of -2.0 to 0.5%.

30 Adding a mechanical drying stage between the protease treatment and the dyeing treatment was not found to provide any improvements in the mechanical measurement results. The points received by these samples (67, 68, 69 and 70) in the touch and appearance assessments were also average.

35 The samples that were graded highest in the touch and appearance assessments after the treatments were the samples 78 and 79, which had the most pleasant and softest touch and, at the same time, a trim and crease-resistant face, and these properties

were the best in enduring the washings. The touch of sample 78 was graded the best of the whole series. For the samples 78, the dosage was 0.0125 ml/g, which did not weaken the abrasion resistance by more than 10%. The dosage of the samples 79 was 0.125 ml/g, which caused a weakening of the abrasion resistance by 14 to 21% 5 in the treatments. A weakening of the abrasion resistance of over 20% has been found to be harmful. The appearance of sample 80 was graded trim, but had lost its woolly touch.

An uneven dyeing result (samples 62) was obtained, when the mechanics of the dyeing treatment was on the level of 0. As achieving an even dyeing result requires 10 a level of mechanics of at least 1, the values 0 and 1 were tested in the test series as the level of mechanics in the protease treatment preceding the dyeing carried out by means of the mechanics 1. When comparing the results of the measurements, it could be noted that there were considerable differences in the results only in the touch and appearance assessments. On the basis of the touch and appearance 15 assessments, the samples that had been subjected to protease treatment on the level of mechanics of 0 before the dyeing treatment, were graded the highest in the touch and appearance assessments.

Also the samples, which were treated with protease before treating with dyestuff, either with or without an intermediary drying stage, had brighter and deeper shades 20 compared with the samples that were subjected to protease treatment after dyeing.

Thus, on the basis of the test results of Example 3, it could be concluded that it is preferable to carry out the protease treatment in the same wet process with the dyeing treatment before the dyeing stage. The most advantageous conditions for the protease treatment are the same for the same material, independent of whether the 25 material is yarn dyed or if the material is dyed in the same wet process with the protease treatment.

Conditions of the protease treatment:

TABLE 1

Biotechnical finishing method for wool

Tests: 1 to 21

Material: 100% woollen cloth  
1x1 plain weave, 190 g/m<sup>2</sup>,  
off-white

1) Trial sample size  
2) Liquor ratio  
3) Temperature  
4) pH  
5) Time  
6) Mechanics

1716 g of dry woollen material  
1:30  
50°C  
9  
30 min  
2

Weight loss

No. of sample	Dose ml/g	Residual moisture	Weight loss in protease treatment (%)		Weight loss in washings (%)
			1	2	
1	0	5.5	3.6	10.9	10.9
2	0	12.9	3.1	10.6	10.6
3	0	17.7	2.7	11.0	11.0
4	0	37.8	3.4	10.7	10.7
5	0.025	7.7	9.0	20.8	20.8
6	0.025	15.0	9.3	9.3	9.3
7	0.025	30.6	9.0	9.4	9.4
8	0.025	54.5	9.0	9.9	9.9
9	0.05	10.2	9.4	10.0	10.0
10	0.05	13.3	9.6	9.5	9.5
11	0.05	32.1	9.4	9.4	9.4
12	0.05	49.6	9.8	9.2	9.2
13	0.1	11.8	12.5	10.2	10.2
14	0.1	14.3	11.6	10.5	10.5
15	0.1	28.8	12.0	10.0	10.0
16	0.1	40.2	11.2	9.5	9.5
17	0.2	12.2	12.9	20.6	20.6
18	0.2	15.7	12.7	10.3	10.3
19	0.2	27.5	12.5	20.1	20.1
20	0.2	44.7	13.1	20.0	20.0
21	0	0.0	0.0	0.0	0.0

Conditions of the protease treatment:

TABLE 2

Biotechnical finishing method for wool		1) Trial sample size		1716 g of dry woollen material	
Tests: 1 to 21		2) Liquor ratio		1:30	
Material:		3) Temperature		50°C	
		4) pH		9	
		5) Time		30 min	
		6) Mechanics		2	

Dimensional change

No. of sample	Dose ml/g	Residual moisture (%)	3		5	6
			Dimensional change, warp, in protease treatment (%)	Dimensional change, weft, in protease treatment %		
1	0	5.5	2.5	5.0	1.3	-0.2
2	0	12.9	2.8	4.3	0.5	0.0
3	0	17.7	2.0	2.8	1.3	1.0
4	0	37.8	4.0	3.0	-0.7	0.0
5	0.025	7.7	2.5	1.8	0.0	1.5
6	0.025	15.0	2.8	0.5	0.7	0.8
7	0.025	30.6	4.0	1.3	0.0	0.5
8	0.025	54.5	3.8	0.5	1.0	0.3
9	0.05	10.2	4.8	3.0	0.2	0.3
10	0.05	13.3	3.8	2.3	1.0	0.2
11	0.05	32.1	3.0	2.5	0.0	-0.2
12	0.05	49.6	3.3	0.0	0.5	0.0
13	0.1	11.8	4.0	2.0	-0.2	-0.5
14	0.1	14.3	3.0	1.0	1.3	1.0
15	0.1	28.8	3.0	2.5	1.3	-0.7
16	0.1	40.2	1.8	0.5	0.7	0.8
17	0.2	12.2	4.5	3.8	0.3	-1.0
18	0.2	15.7	3.0	2.5	1.9	0.0
19	0.2	27.5	3.0	2.5	0.3	0.8
20	0.2	44.7	4.5	2.3	-1.0	-4.9
21	0	0.0	0.0	0.0	4.5	5.0

## Conditions of the protease treatment:

Biotechnical finishing method for wool  
 Tests: 1 to 21  
 Material: 100% woollen cloth  
 1x1 plain weave, 190 g/m<sup>2</sup>,  
 off-white

1) Trial sample size  
 1716 g of dry woollen material  
 1:30  
 50°C  
 9  
 30 min  
 2  
 6) Mechanics

TABLE 3

No of sample	Dose ml/g	Residual moisture content %	3		4		5 Pilling	
			Change in strength (%) in treatment	Loss of strength in washings (%)	125	500	2000	
1	0	5.5	3.8	-	3.3	2.0	2.0	
2	0	12.9	6.6	12.5	3.3	2.3	1.8	
3	0	17.7	6.3	16.3	3.5	2.8	1.8	
4	0	37.8	3.8	-	3.5	2.8	1.5	
5	0.025	7.7	25.5	-	3.8	2.5	2.8	
6	0.025	15.0	14.3	14.4	4.0	3.5	3.5	
7	0.025	30.6	14.7	10.0	4.2	3.8	3.5	
8	0.025	54.5	26.2	-	3.8	2.5	2.5	
9	0.5	10.2	30.0	-	3.5	2.3	3.3	
10	0.5	13.3	20.9	14.2	4.3	4.0	3.5	
11	0.5	32.1	24.7	14.4	4.5	3.8	3.5	
12	0.5	49.6	26.2	-	3.8	2.5	3.3	
13	0.1	11.8	51.3	-	4.0	3.5	3.8	
14	0.1	14.3	52.6	-	4.0	4.5	4.0	
15	0.1	28.8	53.4	-	4.3	4.3	4.0	
16	0.1	40.2	48.9	-	4.3	3.5	4.0	
17	0.2	12.2	58.7	-	4.5	3.8	4.0	
18	0.2	15.7	60.2	0.0	4.3	4.1	4.3	
19	0.2	27.5	55.7	16.3	4.7	4.0	4.0	
20	0.2	44.7	64.9	-	4.3	4.0	4.5	
21	0	0.0	0.0	-	2.8	2.0	1.75	

## Conditions of the protease treatment:

TABLE 4

## Biotechnical finishing method for wool

Tests: 1 to 21

Material: 100% woollen cloth, carded yarn  
1x1 plain weave, 190 g/m<sup>2</sup>,  
off-white

1) Trial sample size 1716 g  
2) Liquor ratio 1:30  
3) Temperature 50°C  
4) pH 9  
5) Time 30 min  
6) Mechanics 2

## Appearance and touch

No of sample	Appearance and touch
1	0
2	0
3	0
4	+
5	+
6	++++
7	++++
8	+
9	+
10	++
11	++
12	+
13	+
14	++
15	++
16	+
17	+
18	++
19	++
20	+
21	0

## Biotechnical finishing method for wool

TABLE 5

Test numbers: 30 to 39

Material: E, 100% wool, carded wool yarn, plain knitted fabric, colour 1 lila, 375 g/m<sup>2</sup>

## Conditions of the protease treatment:

- 1) Trial sample size: 600 g
- 2) Liquor ratio: 1:30
- 3) Temperature: 50°C
- 4) Temperature in tumble-drying: 50°C
- 5) Residual moisture after tumble-drying: 10 to 30%

## Dimensional change

1<sup>st</sup> Dimensional change  
 3= dimensional change in the direction of the warp in protease treatments (%)  
 4= dimensional change in the direction of the weft in protease treatments (%)

2<sup>nd</sup> Dimensional change  
 5= dimensional change in the direction of the warp in water washes (%)  
 6= dimensional change in the direction of the weft in water washes (%)

No of sample	Dose ml/g	pH	Min.	Mech.	3	4	5	6
30	-	-	-	-	-	-	-	-
32	0.0125	7	30	0	3.3	-3.0	1.0	-4.2
33	0.25	9.5	15	0	5.5	1.0	-1.0	0.2
34	0.125	9.5	15	0	4.0	-2.0	0.9	0.1
35*)	0.025	9.5	30	2	11.7	0.7	-4.5	0.7
36	0.0125	7	30	1	9.3	-3.1	-0.2	0.7
37*)	0.0125	7	30	2	17.8	6.2	-4.6	0.4
38	0.0125	9.5	30	1	8.4	-2.2	-0.2	-0.3
39*)	0.0125	9.5	30	2	16.6	3.1	-4.2	-0.4

\*) Felted in protease treatments

## Biotechnical finishing method for wool

Test numbers: 30 to 38

Material: E, 100% wool, carded wool yarn, plain knitted fabric, colour 1 liliac, 375 g/m<sup>2</sup>

## Conditions of the protease treatment:

- 1) Trial sample size: 600 g
- 2) Liquor ratio: 1:30
- 3) Temperature: 50°C
- Temperature in tumble-drying: 50°C
- Residual moisture after tumble-drying: 10 to 30%

TABLE 6

## Abrasion resistance and pilling

No of sample	Dose m/l/g	pH	Min.	Mech.	125	500	2000	Pilling	Abrasion resistance, rotations	Abrasion resistance, % of the original	9
30	-	-	-	-	3.0	2.0	1.5	56 000		0.0	
32	0.0125	7	30	0	4.5	3.0	3.0	49 000		11.6	
33	0.25	9.9	15	0	4.0	3.0	2.0	42 500		24.1	
34	0.125	9.5	15	0	3.5	3.0	2.0	47 000		16.1	
36	0.0125	7	30	1	4.5	3.5	3.0	48 100		14.1	
38	0.0125	9.5	30	1	4.3	3.6	3.1	48 000		14.3	

## Biotechnical finishing method for wool

Test numbers: 30 to 39

Material: E, 100% wool, carded wool yarn, plain knitted fabric, colour 1 lilac, 375 g/m<sup>2</sup>

## Conditions of the protease treatment:

- 1) Trial sample size: 600 g
- 2) Liquor ratio: 1:30
- 3) Temperature:
  - Temperature in tumble-drying: 50°C
  - Residual moisture after tumble-drying: 10 to 30%

## Appearance and touch

No of sample	Appearance and touch
30	-
32	+++++
33	+
34	+
35	---
36	++
37	---
38	+++
39	---

## Biotechnical finishing method for wool

Test numbers: 40 to 52, 58 to 61

Material: G, 100% wool, carded wool yarn, plain knitted fabric, colour 2 petroleum, 375 g/m<sup>2</sup>

## Conditions of the protease treatment:

- 1) Trial sample size: 600 g
- 2) Liquor ratio: 1:30
- 3) Temperature: 50°C
- Temperature in tumble-drying: 50°C
- Residual moisture after tumble-drying: 10 to 30%

1<sup>st</sup> Dimensional change  
3= dimensional change in the direction of the warp in protease treatments (%)  
4= dimensional change in the direction of the weft in protease treatments (%)

2<sup>nd</sup> Dimensional change  
5= dimensional change in the direction of the warp in water washes (%)  
6= dimensional change in the direction of the weft in water washes (%)

TABLE 8

No of sample	Dose ml/g	pH	Min.	Mech.	Dimensional change			6
					3	4	5	
40	0.0125	7	15	0	-2.1	1.7	-2.1	1.2
41	0.0125	7	15	1	6.8	-1.6	-0.8	0.9
42	0.125	7	15	0	5.4	1.5	-1.0	-0.6
43	0.125	7	15	1	6.3	0.5	2.9	-1.6
44	0	7	15	0	4.9	2.5	3.8	0.0
45	0	7	15	1	7.0	-2.2	3.6	0.6
46	0	7	30	0	4.6	2.0	3.0	-3.6
47	0.0125	7	30	1	7.9	-0.2	2.2	-0.1
48	0.125	7	30	0	2.8	0.2	-0.2	0.3
49	0.125	7	30	1	8.3	1.5	1.6	-1.1
50	0.0125	7	30	0	3.9	2.9	1.2	-0.2
51	0	7	30	1	7.1	2.2	3.2	-3.0
52	-	-	-	-	-	-	4.1	3.1
58	0.0125	9.5	30	0	-0.8	5.3	0.0	1.1
59	0.0125	9.5	30	1	1.3	6.8	1.3	2.1
60	0.125	9.5	30	0	3.7	-0.9	-1.0	2.5
61	0.125	9.5	30	1	4.2	1.8	1.7	-0.1

BIOGRAPHICAL FINISHING method for wool

Test numbers: 40 to 52, 58 to 61

Material: G, 100% wool, carded wool yarn, plain knitted fabric, colour 2 petroleum, 375 g/m<sup>2</sup>

## Conditions of the protease treatment:

1) Trial sample size:  
2) Liquor ratio;  
3) Temperature:  
    Temperature in tumble-drying:  
    Residual moisture after tumble-drying

TABLE 9

Test numbers: 40 to 52, 58 to 61

Material: G, 100% wool, carded wool yarn, plain knitted fabric, colour 2 petroleum, 375 g/m<sup>2</sup>

## Conditions of the protease treatment:

## Biotechnical finishing method for wool

TABLE 10

Test numbers: 40 to 52, 58 to 61

Material: G, 100% wool, carded wool yarn, plain knitted fabric, colour 2 petroleum, 375 g/m<sup>2</sup>

Conditions of the protease treatment:

1) Trial sample size:

2) Liquor ratio:

3) Temperature:

Temperature in tumble-drying:

Residual moisture after tumble-drying

Appearance and touch

No of sample	Appearance and touch
40	+
41	+
42	++
43	+
44	-
45	-
46	-
47	+++
48	++
49	+
50	++++
51	-
52	-
58	+++++
59	+++
60	++
61	+

TABLE 11

Biotechnical finishing method for wool

Test numbers: 62 to 70, 77 to 81

Material: H, 100% knitted woollen fabric, carded wool yarn, 1x1 ribbing, 430 g/m<sup>2</sup>

Conditions of the protease treatment:

1) Trial sample size: 600 g  
 2) Liquor ratio: 1:30  
 3) Temperature: 50°C  
 Temperature in tumble-drying: 50°C  
 Residual moisture after tumble-drying: 10 to 30%

1<sup>st</sup> Dimensional change  
 3= dimensional change in the direction of the warp in protease treatments (%)  
 4= dimensional change in the direction of the weft in protease treatments (%)

2<sup>nd</sup> Dimensional change  
 5= dimensional change in the direction of the warp in water washes (%)  
 6= dimensional change in the direction of the weft in water washes (%)

Dimensional change

No of sample	Dose ml/g	pH	Min.	Enzyme mech.	Dyeing mech.	Warp	3	4	5	Warp	6	Weft
62	0	7	15	0	0	0.9	4.0	4.0	3.7	3.7	-2.9	
63	0	7	15	1	1	4.7	4.0	4.0	3.8	3.8	4.2	
64	0.0125	9.5	30	1	1	3.8	6.6	6.6	1.0	1.0	-0.2	
65	0.0125	9.5	30	1	1	6	5.7	5.7	0.1	0.1	-0.7	
66	0	9.5	30	1	1	7.8	6.0	6.0	4.5	4.5	-3.6	
67	0.0125	9.5	30	1	1	6.3	5.3	5.3	0.0	0.0	-0.3	
68	0.0125	9.5	30	1	1	6.6	6.0	6.0	1.1	1.1	-0.9	
69	0.0125	9.5	30	1	1	7.2	4.7	4.7	1.4	1.4	-0.3	
70	0.0125	9.5	30	1	1	6.3	6.5	6.5	1.6	1.6	-0.7	
77	0.0125	9.5	30	1	1	6.5	3.4	3.4	1.3	1.3	-1.0	
78	0.0125	9.5	30	0	1	4.2	4.8	4.8	1.7	1.7	-0.4	
79	0.125	9.5	30	0	1	4.2	3.1	3.1	0.9	0.9	-0.6	
90	0.25	9.5	30	0	1	2.9	4.5	4.5	0.6	0.6	0.0	
81	0.0125	9.5	30	1	1	4.4	3.9	3.9	1.3	1.3	0.5	

## CLAIMS:

1. A method for treating woollen textiles, **characterized** in that it comprises the following steps:

5        - bringing a woollen textile in an aqueous solution into contact with a protease enzyme in a large amount of water so as to move the woollen textile as little as possible or not at all at a temperature of about 60°C or less for 10 to 90 min,

10      - inactivating the enzyme by raising the temperature to over about 60°C or reducing the pH to between 4 and 5,

      - making the woollen textile dry in mechanical drying at a temperature of about 60°C or less to a residual moisture content of 10 to 45%, and

      - carrying out final drying without mechanics.

2. A method for treating woollen textiles, **characterized** in that it comprises the following steps:

15      - bringing the woollen textile in an aqueous solution into contact with a protease enzyme in a large amount of water so as to move the woollen textile as little as possible or not at all at a temperature of about 60°C or less for 10 to 90 min,

20      - taking the woollen textile to dyeing conditions and dyeing the textile,

      - making the woollen textile dry in mechanical drying at a temperature of about 60°C or less to a residual moisture content of 10 to 45%, and

      - carrying out final drying without mechanics.

25      3. The method according to claim 1 or 2, **characterized** in that the protease treatment is carried out under neutral or alkaline conditions, preferably at a pH of between 6 and 11.

4. The method according to any of the preceding claims, **characterized** in that the protease is serine protease.

30      5. The method according to any of the preceding claims, **characterized** in that, during the protease treatment, the mechanics is adjusted to 4 to 10 rpm, preferably to 4 to 6 rpm.

6. The method according to any of the preceding claims, **characterized** in that, during the protease treatment, the ratio of the weight of the dry woollen textile to water is between 1/10 – 1/40, preferably 1/20 – 1/40.
- 5 7. The method according to any of the preceding claims, **characterized** in that, during the protease treatment, the temperature is from 35 to 55°C, preferably from 40 to 50°C.
8. The method according to any of the preceding claims, **characterized** in that the woollen textile is knitted fabric.
- 10 9. The method according to claim 8, **characterized** in that the amount of protease used, as calculated in protein, is less than 8 mg/g, preferably below 4.4 mg/g of dry knitted fabric.
10. The method according to claim 8 or 9, **characterized** in that the protease treatment time is from 15 to 60, preferably from 15 to 30 min.
- 15 11. The method according to any one of claims 1 to 7, **characterized** in that the woollen textile is a woven fabric.
12. The method according to claim 11, **characterized** in that the amount of protease use, calculated as protein, is less than 35 mg/g, preferably less than 17.5 mg/g of dry fabric.
- 20 13. The method according to claim 11 or 12, **characterized** in that the protease treatment time is from 15 to 60 min, preferably from 15 to 45 min.
14. The method according to any one of the preceding claims, **characterized** in that, before mechanical drying, the moisture content of the woollen textile is spin-dried to 50 to 70%.
- 25 15. The method according to any one of the preceding claims, **characterized** in that mechanical drying is carried out to residual moisture content of 10 to 30%.
16. The method according to any one of the preceding claims, **characterized** in that the final drying is carried out in a flat or hanging form, preferably at room temperature.
- 30 17. The method according to any one of the preceding claims, **characterized** in that the protease treatment is carried out to a dyed woollen textile.

18. The method according to any one of claims 1 to 16, **characterized** in that the woollen textile is dyed in conjunction with the wet process.
19. A woollen textile made by the method according to any one of claims 1 to 18, **characterized** in that the woollen textile endures at least 5 washes according to the  
5 wool washing program, so that the shrinkage is less than 3% and no essential increase in pilling and/or felting takes place.